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S. Bajt[a], H. N. Chapman[a], E. Spiller[b], S. Hau-Riege[a] J. Alameda[a] , A. J. Nelson[a], C. C. Walton[a], B. Kjornrattanawanich[c], A. Aquila[d], F. Dollar[d] E. Gullikson[d], C. Tarrio[e]

[a]Lawrence Livermore National Laboratory, 7000 East Avenue, L-210, Livermore, CA 94550, USA,
[b]Spiller X-ray Optics, Livermore CA 94550, USA,
[c]Universities Space Research Association, NSLS Beamline X24C, Upton, New York 11973, USA,
[d]Lawrence Berkeley National Laboratory, Berkeley, California 94720,
[e]NIST, 100 Bureau Drive, Stop 8411, Gaithersburg, MD 20853-8411, USA.

ABSTRACT.

Multilayers are artificially layered structures that can be used to create optics and optical elements for a broad range of x-ray wavelengths, or can be optimized for other applications. The development of next generation x-ray sources (synchrotrons and x-ray free electron lasers) requires advances in x-ray optics. Newly developed multilayer-based mirrors and optical elements enabled efficient band-pass filtering, focusing and time resolved measurements in recent FLASH (Free Electron LASer in Hamburg) experiments. These experiments are providing invaluable feedback on the response of the multilayer structures to high intensity, short pulsed x-ray sources. This information is crucial to design optics for future x-ray free electron lasers and to benchmark computer codes that simulate damage processes.

Keywords: multilayers, optics damage, free electron laser

1. INTRODUCTION

Multilayers represent a special class of microstructures. These periodic structures of usually two alternating materials of differing refractive index are being used as synthetic crystals covering a wide range of wavelengths. In this paper we primarily focus on multilayers used as Extreme Ultraviolet (EUV)/soft X-ray optics components. To achieve the highest reflectivity and optimal mirror performance, the interfaces between the two alternating materials need to be sharp and smooth. The smaller the period (the sum of the two alternating material thicknesses) the tighter the tolerances for these interface imperfections. Emergence of EUV lithography (EUVL) stimulated many scientific and technological breakthroughs in multilayer science and technology that led to better understanding of the deposition processes and the development of more reliable and stable coating tools. These substantially improved the quality of the multilayers and their performance. Multilayers are deposited in vacuum systems most often using magnetron[1,2] or ion beam sputtering[3,4] although excellent mirrors are also obtained by electron beam deposition with subsequent ion beam polishing[5,6]. Molybdenum/silicon pair proved to be the best choice for normal incidence multilayers operating at 13.5 nm, the wavelength of choice for EUVL. However, it took more than 20 years of research and development to achieve nearly perfect multilayers with 70% reflectivity either by minimizing the mixing/interdiffusion on interfaces with thin, diffusion barriers[7,8] or by smoothing the interfaces with ion beam polishing.[9] In Mo/Si multilayers the only further improvement in reflectivity could come from eliminating or substantially reducing the surface oxide that forms when mirrors get exposed to the air. Currently favored capping layers, such as Si and Ru, form thin (~2 nm) and self-terminating oxide layers but since oxide absorbs EUV its presence always reduces the reflectivity.

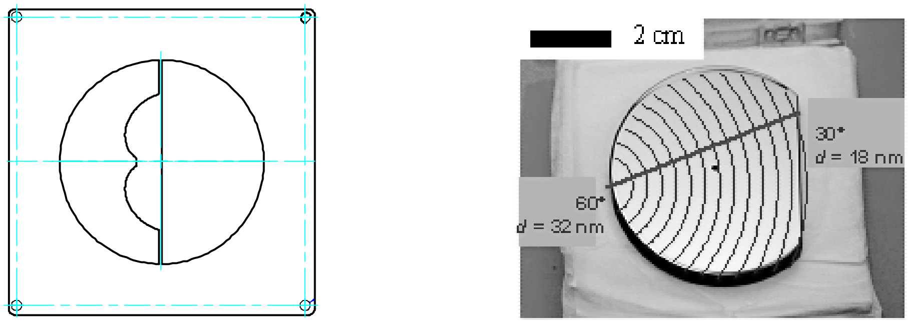
Such multilayer-based optics is used in astronomy to image solar corona,[10,11,12,13] in semiconductor industry for printing very small features,[14,15,16,17] in laser science as diagnostic elements[18] and at synchrotron facilities for use in x-ray microscopy, for example.[19,20,21,22,23] Recently multilayers found a new use in free electron laser (FEL) experiments.[24] Different multilayer-based optical elements enabled efficient band-pass filtering, focusing and time resolution experiments. Multilayers for next generation x-ray sources require more than high reflectivity. These optical elements are exposed to extremely high fluence beams although for very brief times (~100 fs). Therefore the choice of multilayer materials is not governed only by optical but also other properties, such as thermal stability. Currently operating FLASH (Free Electron Laser in Hamburg) at DESY is crucial for learning and evaluating the optics damage and performance. The results are guiding optical designs for future X-ray FELs coming up on-line, such as the Linac Coherent Light Source (LCLS) at SLAC, Stanford, the European X-ray FEL and the Japanese SCSS.

FLASH facility (formerly known as the VUV-FEL) at the Deutsches Elektronen-Synchrotron (DESY) in Hamburg[25] is the first soft X-ray FEL in the world. FLASH generates high power soft X-ray pulses by the principle of self-amplification of spontaneous emission (SASE)[26,27,28,29,30]: a relativistic electron pulse from a superconducting linear accelerator makes a single pass through a periodic magnetic field of an undulator. During the high-gain lasing process, the electrons, perturbed by the magnetic field and by their X-ray photon field, provide the lasing medium. In our experiment, FLASH was operated in a single bunch ultrashort pulse mode[25,31] resulting in 25 fs coherent FEL pulses. The FEL pulses were typically dominated by a single mode, i.e. they were close to transform limited with almost complete transverse and longitudinal coherence. These pulses contained about 10^{12} photons, with a photon wavelength of 32 nm.[25] The peak spectral brilliance of the FLASH soft-X-ray FEL is up to 10^{28} photons/(s mm^2 $mrad^2$ 0.1% bandwidth).[25] This is seven orders of magnitude higher than the peak brilliance of the most advanced synchrotron radiation sources. We had the privilege to be one of the first FLASH users when its 1st harmonic wavelength was at 32 nm. FLASH is a tunable source and our experiments were performed also at 16 nm (2nd harmonic of 32 nm), 13.5 nm (1st harmonic) and 4.5 nm (3rd harmonic of 13.5 nm). Upcoming experiments are planned at even shorter wavelengths (1st harmonic 6 nm, 3rd harmonic at 2 nm).

2. MIRRORS FOR ULTRAFAST COHERENT X-RAY DIFFRACTION EXPERIMENTS

In our first experiments at FLASH we used mirrors operating at 30-32 nm. Although classical multilayer design consists of two alternating materials it has been calculated theoretically[32] and also demonstrated experimentally[33] that for these longer wavelengths a third material in the multilayer structure substantially increases the reflectivity. The multilayer unit structure we developed for 30-32 nm consists of three materials, Si/Mo/B_4C, the order counting from the substrate up. Hence, Si is the first layer deposited on the substrate and B_4C would be the last, top layer in the multilayer. In the initial design we chose Si and B_4C materials based on their high melting temperatures. However, because the mirrors had to reflect uniformly over a wide acceptance angle range (30 to 60 degrees off normal), we had to design a multilayer with large grading in d-spacing. In fact, the d-spacing had to change by a factor of 2 over a length of 28 mm. Since this multilayer structure is under large compressive stress (>1 GPa) peeling off the substrate was of particular concern. Adding Mo as a third material substantially increased the reflectivity but also reduced the stress. Our approach in optimizing the relative thicknesses of Mo, Si and B_4C was based on the design we obtained using IMD[34] software and our knowledge about material properties of these materials. In order to reduce the force due to stress we also minimized the number of layers. This had another positive effect, an increase in the bandwidth of the reflectivity peak.

All mirrors were coated in our magnetron sputtering unit, Mag 3, described in detail elsewhere.[35] The glass substrates, which were 49 mm in diameter were designed by us and fabricated by Research Electro-Optics, Inc. Part of the optic was cut off in order to accommodate the CCD camera (Figure 1). Hence, the length of the optic over which the coating needs to be steeply graded is only ~28 mm long. The steep lateral gradient was achieved with a combination of platter rotation velocity modulation and a shadow mask. A fixed shadow mask in Figure 1 was placed in front of two spinning mirrors, the glass substrate and a witness, Si wafer substrate. The main purpose of these “45 degree” mirrors was to reflect scattered and diffracted light from the exploding samples back to the CCD as shown schematically in Figure 2. The direct FEL beam went through a hole (~150 μm in diameter) in the center of the substrate. This eliminated problems with material ablation, melting and other types of damage due to direct FEL beam and in addition protected the CCD camera. Slightly rougher finish at the outer edge of the hole lowered the reflectivity and scatter of the direct FEL beam back into the CCD and hence increased the signal to noise ratio.



(a) **(b)**

Figure 1: A shadow mask (a) and a coated mirror (b) showing a hole drilled at 45 degree into the substrate.

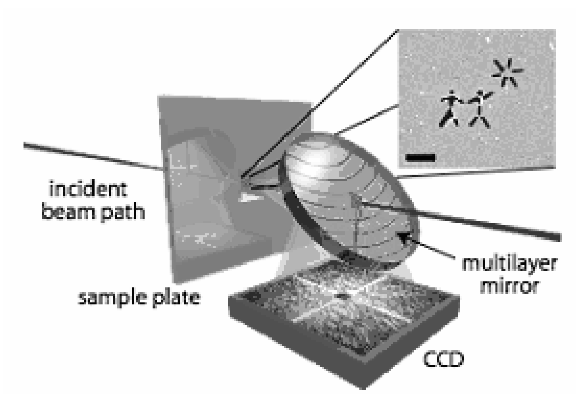
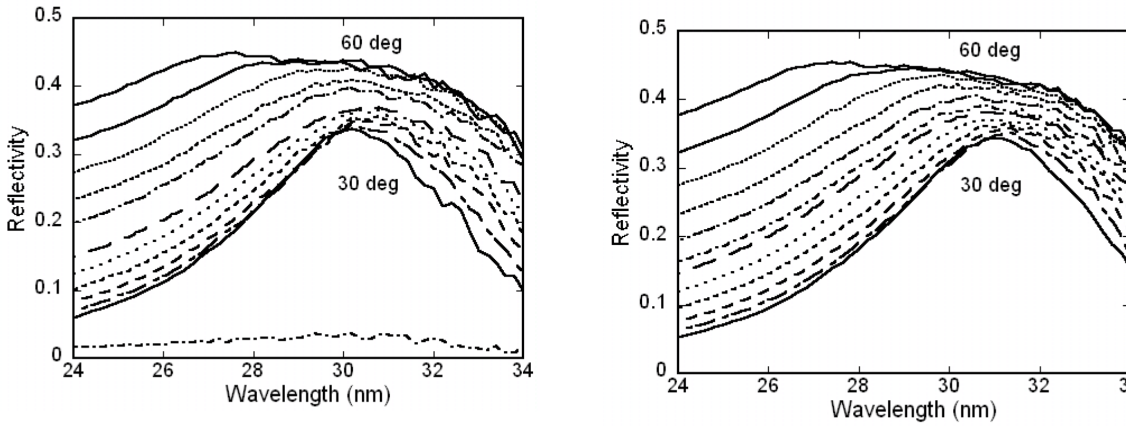


Figure 2: Schematic of the diffraction camera for FLASH, which uses a multilayer-coated mirror to reflect the diffraction pattern onto a CCD. The configuration shown is for the measurement of diffraction from silicon nitride test objects.

Coated mirrors were characterized at the Advanced Light Source[36], National Synchrotron Facility and/or SURF facility. Figure 3 displays 32 nm mirror performance as a function of its position/angle and wavelength. The zero reflectivity curve measured in the center of the optic at 45 degree is due to aforementioned hole (Fig. 3a). We observe no loss in reflectivity due to slightly higher substrate roughness (0.2 nm) of our optic as compared to a multilayer deposited on a super-polished Si wafer substrate, witness sample (Fig. 3b). This is not surprising since substrate roughness and scattering are less detrimental at longer wavelengths. The multilayer reflectivity between 35% (at 30 deg) and 43% (60 deg) was measured on both substrates with multilayer structures consisting of only 10 repeats of Si/Mo/B_4C.

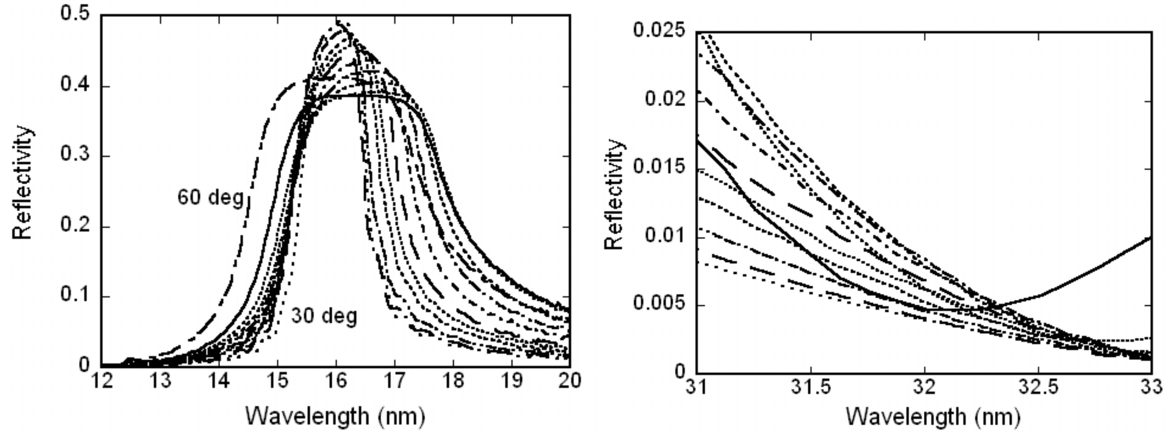
In addition to 32 nm we also developed a multilayer coating to reflect 2nd harmonics at 16 nm. Because of the uncertainty in the exact wavelength of the 1st harmonics it was desirable to design a multilayer with broad bandwidth. The new multilayer consisted of 30 repeats of Si/B_4C/Mo with Si deposited on the substrate first and Mo last. This multilayer gave reflectivity between 40% and 50% at 16 nm (Fig. 4a). In order to suppress reflection of the 1st harmonics at 32 nm we finished the coating with a thick (>40 nm) Si layer. The reflectivity around 32 nm (the 1st harmonic we wanted to suppress) over the whole acceptance angle range was <1% as shown in Figure 4b.

During our second beamtime in winter of 2006 FLASH was optimized for 13.5 nm operation, the same wavelength that EUVL is using. Therefore the “45 degree mirrors” were coated with a standard Mo/Si multilayer design but due to steep lateral gradient and changes in stress only 35 bilayers were used. That was sufficient to achieve 65% (30 deg off normal) to 68% (60 deg off normal) reflectivity as shown in Figure 5.



(a) **(b)**

Figure 3: Measured reflectivity curves of “45 degree” mirror (a) and of a witness (Si wafer) sample. A full reflectivity curve was taken in each point along the central line on the mirror. Each measurement was taken at the incident angle of the experiment in Fig. 2. Measurements at 30 deg (one end) and 60 deg (other end) off normal are labeled on the plots. The measurement at 45 degree shows zero reflectivity in (a) due to the hole in the mirror.



(a) **(b)**

Figure 4: Control “45 degree” mirror for reflecting 2nd harmonics at 16 nm was coated at the same time as the glass substrate mirror (a). Reflectivity between 40 % and 50% was achieved. Antireflective coating was applied to minimize reflectivity at 1st harmonic (32 nm) and the measured reflectivity at 32 nm is <1% (b).

We also developed coatings for 4.5 nm (3rd harmonic of 13.5 nm). This coating consisted of Ni/B_4C/C. Pure Ni/C multilayers with periods between 2.6 and 4.6 nm are too rough due to discontinuous layers of Ni. This phenomenon first observed by Spiller[37] has been studied in more detail. We noticed two effects that point to interface roughness problem: lower reflectivity and an increase in high spatial frequency roughness with the number of layers. As an example, reflectivity curves of a 60 and 120 bilayer Ni/C multilayers are displayed in Figure 6. By adding a thin layer of B_4C on C-on-Ni interface we demonstrated substantial reflectivity increase. Figure 7 shows the reflectivity as a function of B_4C thickness. B^4C thickness of about 0.9 nm seems to give optimum reflectivity performance. For B_4C thickness larger than 0.9 nm no further increase in reflectivity was observed. In fact, the reflectivity starts to be lower because the higher B4C absorption as compared to C outweights the benefit of interface smoothing effect. Better performance could be expected with Co/C multilayers. Hhowever, Co could not be coated with current magnetron setup.

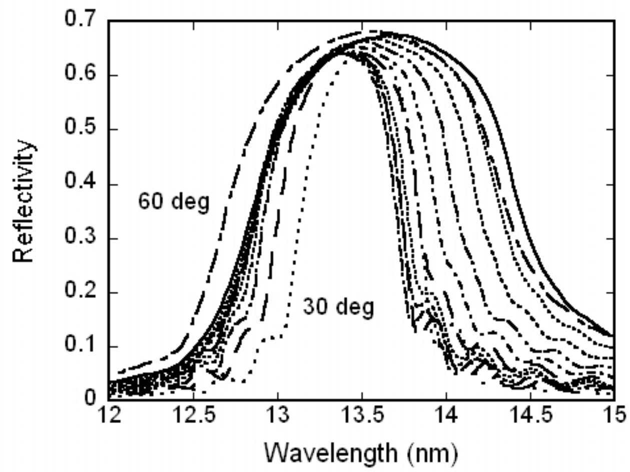


Figure 5: Measured reflectivity curves for 13.5 nm mirror. Reflectivity varies between 65% and 68%.

Wavelength matching is increasingly more challenging for shorter wavelengths due to narrower bandwidths. For example, the coating for 4.5 nm has a reflectivity bandwidth of only 0.1 nm. Measured reflectivity of a "45 degree mirror" is shown in Figure 8 with the optimum performance at 4.6 nm. The reflectivity for this 60 repeat unit of Ni/C/B_4C is between 5% and 11%. The multilayer was terminated with a thicker (>13 nm) C layer that served as an antireflective coating for 13.5 nm (1^{st} harmonic). The reflectivity at 13.5 nm is below 0.04% everywhere on the optic.

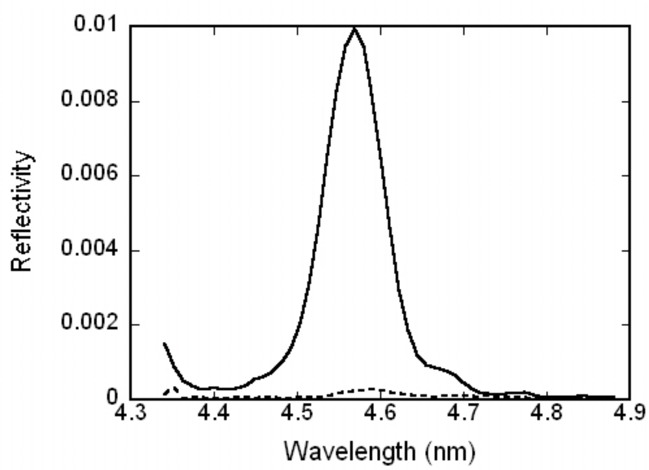


Figure 6: The effect of the number of bilayers on the reflectivity of normal incidence Ni/C multilayers. Experimental reflectivity of a 120 bilayer Ni/C is zero while for a 60 bilayer Ni/C is 1%.

The smallest grazing angles of some of our coating materials are within region of total internal reflection for several of our multilayer materials for λ> 13nm. In that region radiation propagates as an evanescent wave within a film and the phase on reflection does not depend on film thickness. We succeeded to produce coatings with a smooth reflectivity change in this transition region.

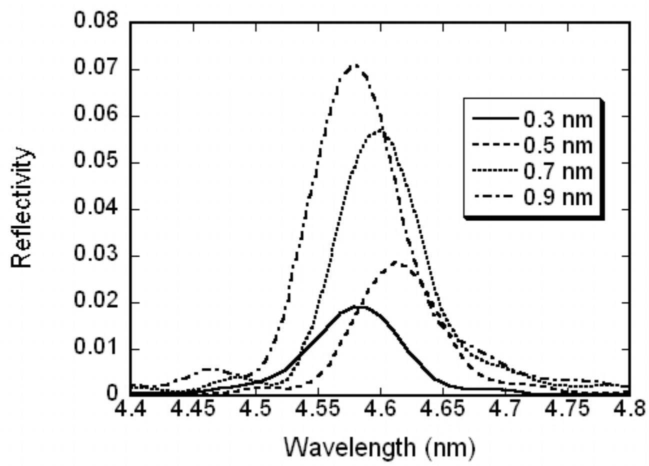


Figure 7: Normal incidence reflectivity as a function of B_4C thickness in Ni/C/B_4C multilayers. All multilayers consisted of 60 repeats of Ni/C/B_4C and had the same Γ ratio (defined as Ni thickness divided by the period thickness). The thickness of B_4C layer partly replacing C layer is displayed in the legend. For B_4C thickness larger than 0.9 nm no further increase in reflectivity was observed. The interfaces could not be smoothed anymore but the effect of higher absorbing B_4C started lowering the reflectivity.

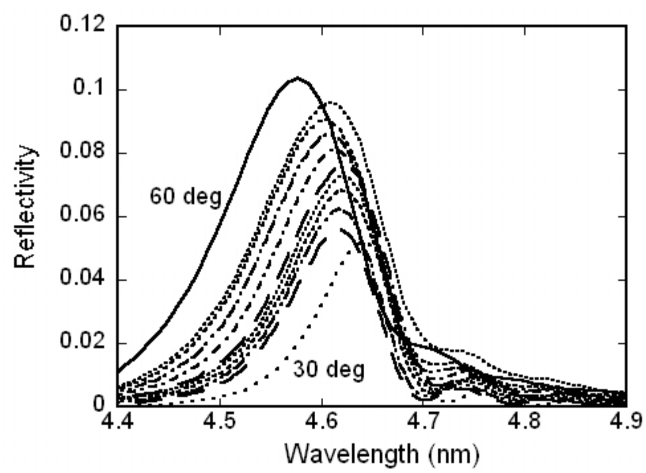


Figure 8: Experimental curves of 45 degree mirror for 4.6 nm wavelength.

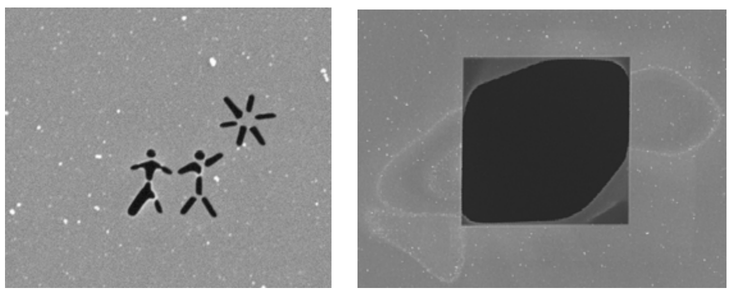
3. OPTICS DAMAGE

3.1. Optics lifetime for EUV lithography application

EUV lithography is based on multilayer coated reflective optics to project the mask image onto the resist-covered wafer. These mirrors consist of 7 nm period Mo/Si multilayers that operate at near normal incidence at 13.5 nm. Initial reflectivity close to 70% is achievable but one of the major unsolved issues is optics lifetime. The EUV mirrors are operating in vacuum. However, since the baking would accelerate the inter-diffusion between Mo and Si and thus reduce the reflectivity and change the alignment of the mirrors, the mirrors are operating in environment with traces of residual water and hydrocarbon vapors. These can adsorb onto the optic surface and be cracked by the energetic (91.8 eV) EUV photons or secondary electrons, producing reactive species that can either oxidize or contaminate the mirror surfaces. Oxidation can be somewhat minimized with protective capping layers. Extended lifetimes have been reported for ruthenium-,[38,39] TiO_2[40]- and carbon-capped multilayer mirrors.[41,42] Some of these materials exhibit self-cleaning surface properties, which could decompose water and degrade organic macromolecules in the presence of UV light. Ideally such capping layers could mitigate both degradation mechanisms of projection optics, carbonization and oxidation. New results on TiO_2 – capped multilayers[43] are very promising.

3.2. Optics damage in FEL applications

Samples (patterns on Si_3N_4 windows, for example) and multilayer optics placed in the focused FEL beam at FLASH are destroyed or severely damaged. An example is shown in Figure 9. A sample on Si_3N_4 membrane (Fig. 9a) is destroyed after one single pulse (Fig. 9b). However, the diffraction pattern of this kind of sample was collected before the damage occured.[24] Multilayer-coated mirrors used in pump-probe and time-holography experiments are also destroyed at the beam location after one single pulse. These, so called single pulse optics are locally destroyed but since the optics is very uniform in wavelength and reflectivity it can be used for many pulses by just moving the optics to another spot. Multilayers proved also very useful for studying optics damage because of their periodic structure and sensitivity to sub-Ångstrom changes in the structure to reflectivity, period thickness and other measurable parameters. As such they are great samples for studying dynamics of materials and testing computer codes in unexplored, high fluence regime. For example, specially designed low reflectivity and narrow bandwidth Si/C multilayers reflecting at 32 nm demonstrated that their reflectivity and the period thickness do not change during 30 fs pulses for fluences of up to 10^{14} W/cm^2.[44,45]



(a) (b)

Figure 9: Sample before (a) and after (b) exposure to the FEL pulse at FLASH.

In contrast, optics placed in the direct, but not focused, FEL beam does not show any damage. An off-axis parabolic mirror, coated with Mo/Si multilayer, was used to focus the FEL beam. The optic was operating at normal incidence angle at 13.5 nm. The reflectivity as a function of position on the optic was measured at the ALS before and after the FEL experiments (Figure 10). Due to the small FEL beam size (~ 2 mm) only the central part of the mirror (± 3 mm) was specified to have correct figure and high finish. Rather low reflectivity of 62% is due to ~0.5 nm rms high spatial frequency roughness of the substrate. Small (~1%) local reduction in reflectivity after the optic was used in FEL experiments is most likely due to surface contamination.

4. SUMMARY

Multilayers are enabling new science at FELs. They are ideal samples to study dynamics of materials under high fluence FEL pulses and to test computational codes that describe the optics damage threshold and high density plasma regime due to their periodic structure that is sensitive to extremely small changes in optical and material properties. We demonstrated successful use of multilayer-based diffraction cameras in FLASH experiments performed at 4.5 nm - 32 nm wavelengths. Optics damage was avoided by letting the direct FEL beam pass through a hole in the optic. Off-axis parabolic mirror coated with Mo/Si multilayer and used with direct FEL beam shows no obvious damage. Hence, optics used in off focus FLASH positions survive relatively intact. In contrast, samples and optics used in the focused FEL beam get destroyed. However, we demonstrated that the information is retrieved before destruction occurs. The solution to such local optics damage is to move the optic in a new location after each pulse. This is the concept of single-pulsed optic which was used for time-delay holography experiments, for example. However, the upcoming XFELs will have at least two orders of magnitude higher intensity than FLASH and only few materials with high melting temperature might be able to withstand XFEL fuences. Nevertheless, multilayer-based optic can be used to increase the reflectivity and angle of grazing incidence mirrors in the experimental chambers since grazing angle and large distance from the source help reduce the optics damage.

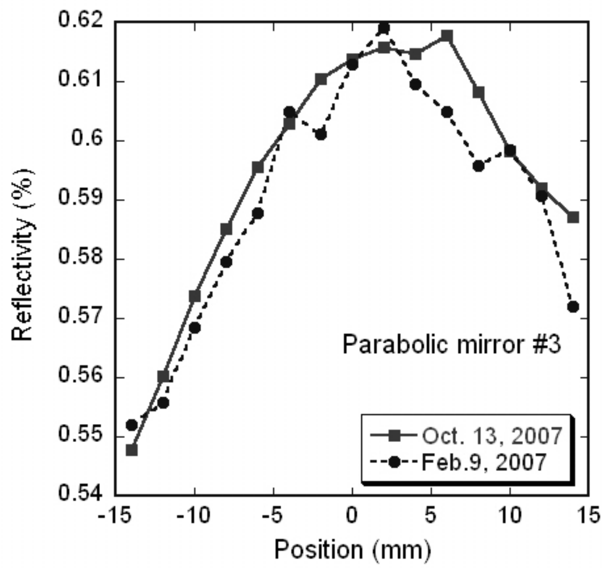


Figure 10: Reflectivity measurements on off-axis parabolic mirror before (squares) and after (circles) using the mirror to focus direct FEL beam.

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